FILE 'USPAT' ENTERED AT 16:25:22 ON 17 JUL 1999

=> set hig off SET COMMAND COMPLETED
L1 QUE (AIRBAG OR GASBAG OR (AIR OR GAS) (W) BAG OR RESTRAINT) AND (149/CLAS OR 280/CLAS)
L2 4769 (AIRBAG OR GASBAG OR (AIR OR GAS) (W) BAG OR RESTRAINT) AND (149/CLAS OR 280/CLAS)
=> set hig on SET COMMAND COMPLETED

L3 18 L2 AND (ZNO OR ZINC (W) PEROXIDE#)
L4 5 L3 AND (5AT OR TETRAZOLE# OR AMINOTETRAZOLE#)

1. 5,883,330, Mar. 16, 1999, Azodicarbonamide containing gas generating composition; Tadao Yoshida, 149/83, 36, 61, 77

2. 5,773,754, Jun. 30, 1998, Gas generating agent with trihydrazino triazine fuel; Yo Yamato, 149/36; 280/741

3. 5,739,460, Apr. 14, 1998, Method of safely initiating combustion of a gas generant composition using an autoignition composition; Gregory D. Knowlton, et al., 102/324, 205; 149/45, 109.6; 280/741

4. 5,525,170, Jun. 11, 1996, Fumaric acid-based gas generating compositions for airbags; Armin Stark, et al., 149/85, 77, 83

5. 5,197,758, Mar. 30, 1993, Non-azide gas generant formulation, method, and apparatus; Gary K. Lund, et al., 280/741; 149/61

US PAT NO: 5,883,330 L4: 1 of 5

BSUM(6) To . . . substitute for sodium azide-based gas generating compositions. For example, Japanese Unexamined Patent Publication No.208878/1991 discloses a composition comprising **tetrazole**, triazole or a metal salt thereof as the main component, an oxygen-containing oxidizing agent such as ammonium perchlorate, sodium nitrate,. . .

BSUM(26) Useful . . . such organic compounds are amino group-containing organic compounds, amido group-containing organic compounds, nitramine group-containing organic compounds, nitrosoamine group-containing organic compounds, **tetrazole** derivatives, etc. Specific examples of amino group- or amido group-containing organic compounds are not critical and include, for example, azodicarbonamide,. . . unlimited and include aliphatic and alicyclic compounds containing one or more nitrosamine groups as substituents, such as dinitrosopentamethylenetetramine (DPT). The **tetrazole** derivatives that can be used are also virtually unlimited and include **aminotetrazole**

BSUM(32) The . . . wide variety of oxide-based decomposition promoters, organic type decomposition promoters, etc. Specific examples of the oxide-based decomposition promoters are CuO, **ZnO**, ZnCO.sub.3, MnO.sub.2, Pb.sub.2 O.sub.3, Pb.sub.3 O.sub.4, PbO.sub.2,. . .

BSUM(36) The . . . of the composition. Useful oxygen generating agents are not specifically limited, and include conventional oxygen generating agents such as CuO.sub.2, **ZnO**.sub.2, etc. The amount of the oxygen generating agent to be used is not essential and, . . .

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BSUM(5) A . . . above-mentioned problems. For example, Japanese Patent Publication No. 57,629/1994 describes a gas generating agent containing a transition metal complex of **tetrazole** or triazole. Further, Japanese Laid-Open (Kokai) No. 254,977/1993 . . .

DETD(4) Examples thereof include **tetrazole** derivatives such as 5-**aminotetrazole**, ditetrazole derivatives, triazole derivatives, dicyanediamide, azodicarbonamide,

DETD(5) A . . . as CuO, Cu.sub.2 O, Co.sub.2 O.sub.3, CoO, Co.sub.3 O.sub.4, Fe.sub.2 O.sub.3, FeO, Fe.sub.3 O.sub.4, MnO.sub.2, Mn.sub.2 O.sub.3, Mn.sub.3 O.sub.4, NiO, **ZnO**,

DETD(18) Theoretical . . . 7 in Table 1. Further, a theoretical burning temperature of a gas generating agent containing a transition metal complex of 5-**aminotetrazole** (5-AT) indicated in Japanese Patent Publication No. 57,629/1994 is shown in Comparative Examples 1 and 2, that of a gas. . . those in Examples 2, 5 and 7. However, in Comparative Examples 1 and 2, low melting burnt residues such as **ZnO** and CuO are melted, which is undesirable. On the other hand, in Examples 2, 5 and 7, only SrO which. . .

DETD(19) . . .
Example 7 5-**aminotetrazole** (10/5/65/20)

US PAT NO: 5,739,460 L4: 3 of 5

ABSTRACT: The . . . nitrite of sodium, potassium, or silver, or a solid organic nitrate, nitrite, or amine, such as guanidine nitrate, nitroguanidine and 5-**aminotetrazole**, respectively. The present invention also relates to a method for initiating a gas generator or pyrotechnic composition in a gas. .
BSUM(13) The . . . nitrite of sodium, potassium, or silver, or a solid organic nitrate, nitrite, or amine, such as guanidine nitrate, nitroguanidine and 5-**aminotetrazole**, respectively.

BSUM(27) In . . . oxides, which include, but are not limited to V.sub.2 O.sub.5, CrO.sub.3, Cr.sub.2 O.sub.3, MnO.sub.2, Fe.sub.2 O.sub.3, Co.sub.3 O.sub.4, NiO, CuO, **ZnO**,

DETD(61) Tetramethyl ammonium nitrate, N(CH.sub.3).sub.4 NO.sub.3, was mixed with 5-**aminotetrazole**, CN.sub.5 H.sub.3, potassium chlorate, KClO.sub.3, and molybdenum, Mo, in accordance with equation XXV, i.e., 11.8% by weight N(CH.sub.3).sub.4 NO.sub.3, 8.2%. . .

DETD(64) Tetramethyl ammonium nitrate, N(CH.sub.3).sub.4 NO.sub.3, was mixed with 5-**aminotetrazole**, CN.sub.5 H.sub.3, potassium perchlorate, KClO.sub.4, and molybdenum, Mo, in accordance with equation XXVI, i.e., 13.1% by weight N(CH.sub.3).sub.4 NO.sub.3, 9.1%. . . and 25.7% by weight Mo. An autoignition temperature of 170.+-.3.degree. C. was determined for this composition by DSC analysis. The 5-**aminotetrazole** used should be anhydrous.

CLMS(4) CLMS(16)

US PAT NO: 5,525,170 L4: 4 of 5

BSUM(6) In . . . mixtures have been proposed (U.S. Pat. No. 4,948,439) containing as their principal component organic compounds rich in nitrogen such as **tetrazoles** or **tetrazole** derivates or tetrazolates. However, the disadvantage of such . . .

BSUM(14) Finally, for the metal oxide, a selection from these groups: Al.sub.2 O.sub.3, B.sub.2 O.sub.3, SiO.sub.2, TiO.sub.2, MnO.sub.2, CuO, Fe.sub.2 O.sub.3, and **ZnO** can be made,

DETD(5) Finally, . . . invention (substance mixture 2) was compared to a further known propellant mixture (substance mixture 1) consisting of 30.8% (by weight) 5-amino-**tetrazole**, 36.1% (by weight) sodium nitrate, and 33.1% (by weight) iron-(III)-oxide (cf. U.S. Pat. . . .

CLMS(2) 2. . . . of a metal oxide selected from the group consisting of Al.sub.2 O.sub.3, B.sub.2 O.sub.3, SiO.sub.2, TiO.sub.2, MnO.sub.2, CuO, Fe.sub.2 O.sub.3, **ZnO**, and mixtures

US PAT NO: 5,197,758 L4: 5 of 5

ABSTRACT: Gas . . . fuel which is a transition metal complex of an aminoarazole. Preferred transition metal complexes are zinc and copper complexes of 5-**aminotetrazole** and 3-amino-1,2,4-triazole, with the zinc complexes most preferred. The propellant compositions also include a conventional oxidizer, such as potassium nitrate. . .

BSUM(15) Non-azide materials, such as **tetrazole** derivatives have also been used in gas generant and explosive compositions. For example, U.S. Pat. No. 1,511,771 discloses

BSUM(31) In accordance with the present invention, the preferred aminoarazole transition metal complexes are zinc and copper complexes of 5-**aminotetrazole** (AT) and 3-amino-1,2,4-triazole (ATr). The Zn(AT).sub.2

DETD(2) The . . . as the non-azide gas producing fuel material. As used herein, the term "aminoarazole" refers to compounds which contain either a **tetrazole** or triazole ring with at least one amino group bonded directly to at least one of the carbon atoms of the **tetrazole** or triazole ring. And 5-**aminotetrazole**

DETD(3) The . . . employed nitrogen producing materials. First, they avoid the aforementioned disadvantages of the azide compounds. Second, while various **aminotetrazoles** . . .

DETD(8) 5[Zn(CH.sub.3 N.sub.5).sub.2]+7[Sr(NO.sub.3).sub.2
.fwdarw.32(N.sub.2)+10(CO.sub.2)+3(H.sub.2 O)+5(**ZnO**)+7[Sr(OH.sub.2)]

L5 13 L3 NOT L4
L6 1 L5 AND (NONAZIDE# OR NON (W) AZIDE#)
L7 12 L5 NOT L6

3. 5,756,928, May 26, 1998, Spontaneously-firing explosive composition; Yuji Ito, et al., 149/7; 102/205; 149/3, 19.1, 19.2, 19.4, 19.5, 19.7, 19.91, 83; 280/741

4. 5,726,382, Mar. 10, 1998, Eutectic mixtures of ammonium nitrate and amino guanidine nitrate; Robert S. Scheffee, et al., 149/19.91, 47, 62

5. 5,656,793, Aug. 12, 1997, Gas generator compositions; Koji Ochi, et al., 149/22, 36, 37, 61, 75, 108.2

7. 5,462,306, Oct. 31, 1995, Gas generator for vehicle occupant restraint; Eric S. Barcaskey, 280/736; 149/35; 280/741

11. 3,950,263, Apr. 13, 1976, Gas cooling and filtering agent for air bag gas generator; Daizo Fukuma, et al., 252/193; 55/522, DIG.33; 280/741; 422/167

L8 7 L7 AND PEROXIDE#

1. 5,898,126, Apr. 27, 1999, Air bag gas generating composition; Tadao Yoshida, 149/46; 102/288; 149/35, 79, 83

2. 5,827,996, Oct. 27, 1998, Air bag gas generating composition; Tadao Yoshida, et al., 149/45, 37, 61, 83, 109.2

3. 5,536,339, Jul. 16, 1996, Air bag inflator gas compositions and inflator containing the same; V. R. Pai Verneker, 149/19.5, 36, 77, 83; 280/728.1, 741

4. 5,216,199, Jun. 1, 1993, Lead-free primed rimfire cartridge; Robert K. Bjerke, et al., 102/471, 204, 443; 149/61, 68

5. 4,834,818, May 30, 1989, Gas-generating composition; Takashi Kazumi, et al., 149/35, 17

6. 4,376,002, Mar. 8, 1983, Multi-ingredient gas generators; Lechoslaw A. M. Utracki, 149/35, 40

7. 3,862,866, Jan. 28, 1975, GAS GENERATOR COMPOSITION AND METHOD; Hubert G. Timmerman, et al., 149/21, 77, 85, 113; 252/183.13, 187.31; 280/741; 422/164

US PAT NO: 5,898,126 L8: 1 of 7

BSUM(17) Japanese . . . such as azodicarbonamide, trihydrazinotriazine or the like and an oxidizing agent such as potassium permanganate, manganese dioxide, barium dichromate, barium **peroxide** or the like. However, the use of potassium permanganate as the oxidizing agent involves the problem of low heat stability. . . the oxidizing agent does not insure satisfactory shock sensitivity or burning velocity, while the use of barium dichromate or barium **peroxide** as the oxidizing agent gives rise to toxic substances

BSUM(37) The . . . and organic polymers. The oxides, chlorides, carbonates and sulfates of the Period 3, Period 4 and Period 6 elements include **ZnO**, ZnCO.sub.3, MnO.sub.2, FeCl.sub.3, CuO, Pb.sub.3 O.sub.4, PbO.sub.2, PbO, Pb.sub.2 O.sub.3, S, TiO.sub.2, V.sub.2 O.sub.5, CeO.sub.2, Ho.sub.2 O.sub.3, CaO.sub.2, Yb.sub.2 O.sub.3,. . .

DETD(11)CaO.sub.2 : calcium **peroxide**
DETD(14)**ZnO**: zinc oxide

DETD(17) TABLE 1
No.
Gas generating base (wt %)
11 ADCA 30 KClO.sub.4 10 CaO.sub.2 60
12 ADCA 41 KNO.sub.3 59 --
13 ADCA (30% **ZnO**) 47 KNO.sub.3 53 --
ADCA (30% **ZnO**): an ADCA--**ZnO** mixture containing 30 wt. % of **ZnO**

DETD(50) . . .
13 HMX 48 NH.sub.4 NO.sub.3 52 -- -- 2 3
14 ADCA 42 KClO.sub.4 58 **ZnO** 4.7 32 34
15 ADCA 45 KClO.sub.4 55 **ZnO** 5 25 32
16 ADCA 45 KClO.sub.4 55 -- -- 6 8 . . .

US PAT NO: 5,827,996 L8: 2 of 7

BSUM(25) The . . . Preferred are those capable of generating and/or feeding oxygen at high temperatures, for example, oxohalogen acid salts, nitrates, nitrites, metallic **peroxides**, hyperoxides,

BSUM(36) Specific . . . tin oxide, zinc oxide and chromium oxide are preferred, and CuO, CoO, NiO, Ni.sub.2 O.sub.3, MoO.sub.3, Cr.sub.2 O.sub.3, TiO.sub.2, SnO, **ZnO** and Fe.sub.2 O.sub.3 . . .

BSUM(39) Among . . . compounds which form MoO.sub.3 when heated, oxygen-containing tungsten compounds which form WO.sub.3 when heated, cobalt phosphomolybdate, Cr.sub.2 .sub.3, TiO.sub.2, SnO, **ZnO**, Fe.sub.2 O.sub.3, etc. are particularly preferred, and . . .

CLMS(1) . . . agent selected from the group consisting of oxohalogen acid salts nitrites, metallic **peroxides**

US PAT NO: 5,536,339 L8: 3 of 7

ABSTRACT: Air . . . The gas generating compositions include lithium, potassium and sodium perchlorates,

optionally with a nitride or non-halogenated polymer or both, styrene **peroxides**, polystyrene **peroxides**, **zinc** **peroxide** in hydrated form, iron oxalate hydrazinate, and iron nitrate hydrazinate

BSUM(14) These . . . and flame upon activation, and at least one gas generating compound selected from the group consisting of LiClO.sub.4, NaClO.sub.4, styrene **peroxide**, polystyrene **peroxide**, hydrated **zinc** **peroxide**, iron oxalate hydrazinate, and iron nitrate hydrazinate, disposed in said second chamber

BSUM(16) and . . . the bag, the improvement which comprises: said gas generating compound is selected from the group consisting of LiClO.sub.4, NaClO.sub.4, styrene **peroxide**, polystyrene **peroxide**, hydrated **zinc** **peroxide**, iron oxalate hydrazinate, and iron nitrate hydrazinate:

DETD(18) A further embodiment of the present invention is the use of **zinc** **peroxide** in hydrated form (**ZnO**.sub.2.1/2 H.sub.2 O) as the gas generating compound. Hydrated **zinc** **peroxide** will explode at 212.degree. C. in the presence of zinc metal. The degradation products are zinc oxide (**ZnO**)

DETD(20) All . . . the chemical arts and most are commercially available. For example a synthesis scheme for making the perchlorate compounds, the styrene **peroxide** and polystyrene **peroxide** compounds, and hydrated **zinc** **peroxide** compounds is described in Encyclopedia of Explosives and Related Items,

US PAT NO: 5,216,199 L8: 4 of 7

BSUM(7) Other . . . No. 4,363,679 to Hagel et al. The Hagel et al. formulation has a smokeless propellant, a titanium fuel, and a **zinc** **peroxide** oxidizer. Another primer formulation using dinol is disclosed in U.S. Pat. No. 4,608,102 to Krampen et al., which uses manganese. . .

US PAT NO: 4,834,818 L8: 5 of 7

BSUM(6) For . . . catalyst. M represents a hydrazino radical, ammonium radical, alkali metal, or alkaline earth metal, and the oxidizer is a metal **peroxide**, inorganic perchlorate, or metal nitrate.

BSUM(14) The . . . the invention is not achieved by the other kinds of solder glass represented by PbO.B.sub.2 O.sub.3, P.sub.2 O.sub.5.Al.sub.2 O.sub.3, B.sub.2 O.sub.3.**ZnO**, PbO.**ZnO**.B.sub.2 O.sub.3, B.sub.2 O.sub.3.**ZnO**.BaO, PbO.B.sub.2 O.sub.3.TiO.sub.2, B.sub.2 O.sub.3.P.sub.2

US PAT NO: 4,376,002 L8: 6 of 7

BSUM(4) The . . . the burn rate or reaction time. In some cases the metal oxide is replaced by a metallic chloride, nitrate, sulfate, **peroxide**, perchloride or other oxidizer. A wide range of these selected combinations are to be found in the patent literature. (See,. . .

BSUM(9) For . . . may optionally contain a minor amount of a further metal oxide selected from the group of TiO.sub.2, Al.sub.2 O.sub.3 and **ZnO** or mixtures of these.

BSUM(13) . . .

M	8/3	MnO.sub.2
CA	4/3	SiO.sub.2
TA	10/1	Ta.sub.2 O.sub.5
Z	4/3	**ZnO** . . .

US PAT NO: 3,862,866 L8: 7 of 7

DETD(11) Other oxygen bearing oxidizing powders are technically feasible for practice of this invention such as; for example, **peroxides**, superoxides, permanganates, nitrates and the like. Permanganates and nitrates are undesirable because of possible toxity and **peroxides** and superoxides are undesirable because

DETD(29) Thus, for example, catalysts may also be selected from the following group: barium **peroxide**, bismuth trioxide, bismuth pentoxide, calcium ferrite, calcium **peroxide**, calcium oxide peroctahydrate, ferroscoferric oxide, hydrated ferric oxide, magnesium **peroxide**, manganese dioxide, manganese sequioxide, manganese (II, III) oxide (Mn.sub.3 O.sub.4), molybdenum oxides, potassium **peroxide**, potassium trioxide, potassium superoxide, sodium **peroxide**, tungsten trioxide and **zinc** **peroxide**.

L9 71 L2 AND PEROXIDE#
L10 25 L9 AND (NONAZIDE# OR NON (W) AZIDE# OR 5AT OR AMINOTETRAZOLE# OR TETRAZOLE#)
L11 25 L10 NOT L8
L12 23 L11 AND (PERCHLORATE# (P) NITRATE#)

1. 5,889,161, Mar. 30, 1999, N,N'-azobis-nitroazoles and analogs thereof as igniter compounds for use in energetic compositions; Jeffrey C. Bottaro, et al., 534/551; 149/55, 56, 88, 109.6

2. 5,847,315, Dec. 8, 1998, Solid solution vehicle airbag clean gas generator propellant; Arthur Katzakian, Jr., et al., 149/19.91, 19.1, 36, 46

3. 5,756,929, May 26, 1998, **Nonazide** gas generating compositions; Norman H. Lundstrom, et al., 149/22, 36, 45, 61, 75, 77

4. 5,735,118, Apr. 7, 1998, Using metal complex compositions as gas generants; Jerald C. Hinshaw, et al., 60/219; 149/45; 280/741

5. 5,725,699, Mar. 10, 1998, Metal complexes for use as gas generants; Jerald C. Hinshaw, et al., 149/19.1, 45, 75; 280/741

6. 5,682,014, Oct. 28, 1997, Bitetrazoleamine gas generant compositions; Thomas K. Highsmith, et al., 149/36, 18, 26, 37, 46, 61, 77, 109.2; 280/741

7. 5,551,725, Sep. 3, 1996, Vehicle airbag inflator and related method; Christopher P. Ludwig, 280/737; 102/531; 222/3; 280/741; 422/166

8. 5,518,054, May 21, 1996, Processing aids for gas generants; Scott C. Mitson, et al., 149/35; 102/287, 290

9. 5,516,377, May 14, 1996, Gas generating compositions based on salts of 5-nitraminotetrazole; Thomas K. Highsmith, et al., 149/18, 19.1, 36, 62, 92, 109.2

10. 5,501,823, Mar. 26, 1996, Preparation of anhydrous **tetrazole** gas generant compositions; Gary K. Lund, et al., 264/3.1; 149/109.6

11. 5,500,059, Mar. 19, 1996, Anhydrous 5-**aminotetrazole** gas generant compositions and methods of preparation; Gary K. Lund, et al., 149/19.1, 61, 70, 77, 109.2, 109.6

12. 5,472,647, Dec. 5, 1995, Method for preparing anhydrous **tetrazole** gas generant compositions; Reed J. Blau, et al., 264/3.1; 149/19.92, 109.6; 264/3.4

13. 5,472,534, Dec. 5, 1995, Gas generant composition containing non-metallic salts of 5-nitrobarbituric acid; Robert B. Wardle, et al., 149/36, 109.6; 280/741; 544/301

14. 5,460,668, Oct. 24, 1995, **Nonazide** gas generating compositions with reduced toxicity upon combustion; Lyman R. Lyon, 149/36, 61

15. 5,439,537, Aug. 8, 1995, Thermite compositions for use as gas generants; Jerald C. Hinshaw, et al., 149/22, 37; 422/165

16. 5,429,691, Jul. 4, 1995, Thermite compositions for use as gas generants comprising basic metal carbonates and/or basic metal nitrates; Jerald C. Hinshaw, et al., 149/45, 22, 37; 280/728.1

17. 5,197,758, Mar. 30, 1993, **Non**-**azide** gas generant formulation, method, and apparatus; Gary K. Lund, et al., 280/741; 149/61

18. 5,160,386, Nov. 3, 1992, Gas generant formulations containing poly(nitrito) metal complexes as oxidants and method; Gary K. Lund, et al., 149/88, 19.5, 35, 45, 109.4

19. 5,139,588, Aug. 18, 1992, Composition for controlling oxides of nitrogen; Donald R. Poole, 149/61, 77, 83

20. 5,125,684, Jun. 30, 1992, Extrudable gas generating propellants, method and apparatus; Richard V. Cartwright, 280/736; 149/19.7, 19.8, 64, 69, 79, 80, 88, 92, 93; 264/3.3, 3.4; 280/741

21. 5,035,757, Jul. 30, 1991, Azide-free gas generant composition with easily filterable combustion products; Donald R. Poole, 149/46, 45, 61, 70, 75, 76, 77, 83, 85

22. 4,758,287, Jul. 19, 1988, Porous propellant grain and method of making same; John F. Pietz, 149/2; 102/291, 292; 149/35, 61, 77, 109.6; 264/3.1; 280/728.1, 741

23. 4,604,151, Aug. 5, 1986, Method and compositions for generating nitrogen gas; Gregory D. Knowlton, et al., 149/35, 76; 252/183.14; 280/736, 741; 422/164

US PAT NO: 5,889,161 L12: 1 of 23

BSUM(8) The . . . potassium permanganate, followed by nitration of the intermediate so formed. U.S. Pat. No. 5,472,647 to Blau et al. describes substituted **tetrazoles** as gas generants. Examples of the **tetrazoles** are bicyclic compounds having the structure ##STR2## in which X, R.sub.1 and R.sub.2 represent H, methyl, ethyl, cyano, nitro, amino,. . . Poole and 5,531,941 to Poole et al., which describe compositions for inflating automobile airbags. The latter compositions may contain nitrated **tetrazoles**; Poole et al. '775 mentions 5-nitrotetrazole and 5-nitroaminotetrazole, while Poole '757 mentions 3-nitro-1,2,4-triazole-5-one. Both patents disclose the use of metal. . .

BSUM(20) Particularly preferred compounds are wherein two five-membered nitrogen-containing heterocycles, e.g., triazoles or **tetrazoles**, are linked to each other through an azo bond. The heterocycles preferably contain at least one nitro substituent bound to. . .

BSUM(40) Gas-generating . . . art. Examples of oxidizers that may be incorporated into the present gas-generating compositions include, but are not limited to, ammonium **nitrate** (AN), phase-stabilized ammonium **nitrate** (PSAN), ammonium dinitramide (ADN), potassium **nitrate** (KN), potassium dinitramide (KDN), sodium **peroxide** (Na.sub.2 O.sub.2), ammonium **perchlorate** (AP),

DETD(14) Synthesis of 1,1'-Azobis-(3-Nitro-1,2,4-**tetrazole**):

DETD(15) The procedure of Example 1 may be repeated using 1-amino-3-nitro-1,2,4,5- **tetrazole** as a starting material, to prepare 1,1'-azobis-(3-nitro-1,2,4,5-**tetrazole**).

DETD(30) A solid composition for generating gases comprising 1,1'-azobis-(3-nitro- 1,2,4-triazole) and an oxidizer chosen from ammonium **nitrate** (AN), ammonium **perchlorate** (AP), and ammonium dinitramide (ADN) is formed into a tablet. The tablet is ignited by either elevating the ambient temperature. . .

DETD(33) A solid composition for use as a propellant comprising 1,1'-azobis-(3-nitro-1,2,4-triazole) and an oxidizer chosen from ammonium **nitrate** (AN), ammonium **perchlorate** (AP), and ammonium dinitramide (ADN), a binder such as R-45M or GAP, with isocyanate curing agents is formed into a. . .

US PAT NO: 5,847,315 L12: 2 of 23

BSUM(9) In order to avoid the problems associated with azides, various **non**-**azide** systems have appeared in the prior art. Hinshaw and Blau described in U.S. Pat. No. 5,439,537 the use of thermite. . . U.S. Patents, for example, U.S. Pat. Nos. 5,472,647, 5,460,668, 5,035,757 and 4,369,079, described the use of azole compounds such as **aminotetrazole**, **tetrazole**, bitetrazole, as well as. . .

BSUM(17) In . . . a polybutadiene with silylferrocene groups. The plasticizer also serves as a burning rate modifier. Their formulations consist generally of ammonium **perchlorate** and sodium **nitrate** as . . .

BSUM(19) Sumrall et al. in U.S. Pat. No. 5,411,615 described the use of a four component eutectic consisting of dicyandiamide, ammonium **nitrate**, guanidine **nitrate** and ethylene diamine dinitrate as a bonding agent for an insensitive high explosive. The ingredients of the explosive, aluminum, RDX, and ammonium **perchlorate** were added to the liquified eutectic mixture at 185.degree. F. in the mixer.

BSUM(21) Yet . . . by Klunsch et al in U.S. Pat. No. 3,926,696. Various multicomponent eutectics, an example of which consists of 11% ammonium **nitrate**, 45% ethanolamine **nitrate**, 16% methylamine **nitrate**, 16% methylamine **perchlorate** and 12% urea, were used to formulate explosives which remain liquid below -10.degree. C. An example of such an explosive contained 52.5% ammonium **nitrate**, 3% sodium **nitrate**, 22.5% of the eutectic mixture and 22% aluminum. The eutectic served to keep ingredients

BSUM(35) In . . . propellants burn at ambient temperature and pressure. Propellants containing these polyethers polymers and a burning rate catalyst such as chromium 5-**aminotetrazole** complex (CrATZ) burn more vigorously when ignited than propellants without this polymer. With or without this polymeric additive

BSUM(43) In . . . and were oxygen balanced with the eutectic oxidizers to produce water, carbon dioxide, and nitrogen gases. Other additives such as 5-**aminotetrazole** nitrate, urea nitrate, and equivalent compounds may also be used in these formulations as combustion modifiers.

US PAT NO: 5,756,929 L12: 3 of 23

BSUM(9) More specifically, the present invention comprises the use of one or more high nitrogen **nonazides** selected from the group consisting of nitroguanidine, nitroaminoguanidine, guanidine **nitrate**, guanidine **perchlorate**, guanidine picrate, cyanuric hydrazide, and diammonium bitetrazole, alone or in combination with other high nitrogen **nonazides**, such as **tetrazoles**, bitetrazoles,

BSUM(13) The foregoing preferred primary high nitrogen **nonazide** fuels can be suitably combined with other known secondary high nitrogen **nonazide** fuels without sacrificing the benefits resulting from their use. The secondary high nitrogen **nonazide** fuels which can be combined with the preferred primary high nitrogen **nonazide** guanidine, triazine, and **tetrazole** fuels specifically discussed above, include other guanidine compounds such as the metal salts of nitroaminoguanidine, metal salts of nitroguanidine, nitroguanidine **nitrate**, nitroguanidine **perchlorate**, **tetrazoles** such as 1H-**tetrazole**, 5-**aminotetrazole**, 5-nitrotetrazole, 5-nitroaminotetrazole, 5,5'-bitetrazole, diguanidinium-5,5'-azotetrazolate,

BSUM(15) The foregoing guanidines, alone or in combination with other known high nitrogen **nonazides**, are generally employed in combination with an oxidizer, which is designed to supply most if not all of the oxygen required for combustion. Suitable oxidizers are known in the art and generally comprise inorganic nitrites, **nitrates**, chlorites, chlorates, **perchlorates**, oxides, **peroxides**, persulfates, chromates, and perchromates. Preferred oxidizers are alkali metal and alkaline earth metal **nitrates**, chlorates, **perchlorates** such as strontium **nitrate**, potassium **nitrate**, sodium **nitrate**, barium **nitrate**, potassium chlorate, potassium **perchlorate** and mixtures thereof. . . .

DETD(2) In Examples 1 to 9 the compositions of the present invention are compared to the prior art compositions based on 5-**aminotetrazole** (Example 1, Table 1) as the sole **nonazide** fuel. . . .

DETD(3) TABLE 1
EXAMPLES 1 2 3 4 5
5-**aminotetrazole** 28.60 16.19 11.29 14.30 9.53

DETD(25) A mixture of 5-**aminotetrazole** (**5AT**), guanidine nitrate, and strontium nitrate was prepared having the following composition in percent by weight: 25.00% **5AT**, 25.00% guanidine nitrate, and 50.00% strontium nitrate. These powders were ground separately and dry blended.

DETD(79) A mixture of guanidine **nitrate**, 5-**aminotetrazole**, potassium **perchlorate**, and strontium **nitrate** was prepared having the following composition in percent by weight: 19.90% guanidine **nitrate**, 22.40% 5-**aminotetrazole**, 14.70% potassium **perchlorate**, and 43.00% strontium **nitrate**. These powders were ground separately and dry blended. . . .

DETD(91) . . . 1 2 3 4 5 6 7 8 9 10
5-**Aminotetrazole** 28.60 25 16.19 . . .

DETD(93) Even when the 5-**aminotetrazole** fuel of the stoichiometric baseline **nonazide** composition is only partially substituted with guanidine nitrate (Examples 2, 3, 4 and 5 of Table 1), a significant increase. . . also accomplished by substituting nitroguanidine alone (Examples 1-5 of Table 3) or in combination with guanidine nitrate for the baseline **aminotetrazole** component (Examples 17 and 18). Again a significant improvement in gas yield results at slightly higher but acceptable flame temperatures.. . .

DETD(96) Example . . . evaluated with strontium nitrate as the oxidizer provides a fuel that yields a gas mass fraction at comparable temperature to 5-**aminotetrazole**.

CLMS(5) 5. The gas generant composition of claim 4 wherein the oxidizer is an alkali metal, alkaline earth metal, or transition metal **nitrate**, nitrite, chlorate, chlorite, **perchlorate**, ... mixtures thereof.

US PAT NO: 5,735,118 L12: 4 of 23

ABSTRACT: Gas . . . the complex combusts, nitrogen gas and water vapor is produced. Specific examples of such complexes include metal nitrite ammine, metal **nitrate** ammine, and metal **perchlorate** ammine complexes, as well as hydrazine complexes. A binder and co-oxidizer . . .

BSUM(19) The . . . anions are provided to balance the charge of the complex. Examples of typical oxidizing anions which can be used include **nitrates**, nitrites, chlorates, **perchlorates**, **peroxides**, and superoxides. In some cases the oxidizing anion is part of the metal cation coordination complex.

BSUM(26) As . . . is part of the coordination complex with the metal cation. Examples of typical oxidizing anions which can be used include **nitrates**, nitrites, chlorates, **perchlorates**, **peroxides**, and

BSUM(30) Complexes which fall within the scope of the present invention include metal **nitrate** ammines, metal nitrite ammines, metal **perchlorate** ammines, metal nitrite hydrazines, metal **nitrate** hydrazines, metal **perchlorate** hydrazines, and mixtures thereof. Metal ammine complexes are defined as coordination complexes including ammonia as the coordinating ligand. The ammine complexes can also have one or more oxidizing anions, such as nitrite (NO.sub.2.sup.-), **nitrate** (NO.sub.3.sup.-), chlorate (ClO.sub.3.sup.-), **perchlorate** (ClO.sub.4.sup.-), **peroxide** (O.sub.2.sup.2-), ... or mixtures thereof,

BSUM(64) It . . . or aluminum, carbon, silicon, titanium, zirconium, and other similar conventional fuel materials, such as conventional organic binders. Oxidizing species include **nitrates**, nitrites, chlorates, **perchlorates**, **peroxides**, and other similar oxidizing materials. . . .

BSUM(79) The . . . the oxides of nitrogen from the combustion products of a gas generant composition, including alkali metal salts and complexes of **tetrazoles**, **aminotetrazoles**, triazoles and related nitrogen heterocycles of which potassium **aminotetrazole**, sodium carbonate and potassium carbonate are exemplary. The composition can also include materials which facilitate the release of the composition. . .

US PAT NO: 5,725,699 L12: 5 of 23

ABSTRACT: Gas . . . the complex combusts, nitrogen gas and water vapor is produced. Specific examples of such complexes include metal nitrite ammine, metal **nitrate** ammine, and metal **perchlorate** ammine complexes, as well as hydrazine complexes. A binder and co-oxidizer can be combined with the metal

US PAT NO: 5,682,014 L12: 6 of 23

ABSTRACT: A solid composition for generating a nitrogen containing gas is provided. The composition includes an oxidizer and a **non**-**azide** fuel selected from a bitetrazoleamine or a derivative . . .

BSUM(15) One group of chemicals that has received attention as a possible replacement for sodium azide includes **tetrazoles** and triazoles. These materials are generally coupled with conventional oxidizers such as KNO.sub.3 and Sr(NO.sub.3).sub.2. Some of the **tetrazoles** and triazoles that have been specifically mentioned include 5-amino-**tetrazole**, 3-amino-1,2,4-triazole, 1,2,4-triazole, 1H-**tetrazole**,

DETD(6) In . . . flame temperature and an improved filterable slag. Such oxidizers include metal oxides and metal hydroxides. Other oxidizers include a metal **nitrate**, a metal nitrite, a metal chlorate, a metal **perchlorate**, a metal **peroxide**, ammonium **nitrate**, ammonium **perchlorate** and the. . .

CLMS(10) 10. . . . generating composition according to claim 1 which also includes a secondary oxidizer selected from the group consisting of a metal **nitrate**, a metal nitrite, a metal **peroxide**, a metal carbonate, a metal chlorate, a metal **perchlorate**, ammonium **nitrate**, and ammonium **perchlorate**.

US PAT NO: 5,551,725 L12: 7 of 23

DETD(16) Energetic . . . (where it meets the thermal and age stability requirements for the inflator and its application), etc. Other energetic fuels include **tetrazole** derivatives (e.g., 5-**aminotetrazole**), . . .

DETD(23) Solid oxidizers useful as components of the gas producing source include ammonium **nitrate**, metal chlorates and **perchlorates** (e.g., potassium **perchlorate**, sodium **perchlorate**, lithium **perchlorate**, etc.), metal **nitrates** (e.g., potassium **nitrate**, sodium **nitrate**, etc.), metal oxides (e.g., manganese dioxide, cupric oxide, ferric oxide, etc.), and metal **peroxides** (e.g., calcium **peroxide**, barium **peroxide**, etc.). Preferred solid oxidizers include

US PAT NO: 5,518,054 L12: 8 of 23

BSUM(10) The . . . desire to get away from the use of azide fuels and a number of other fuels have been proposed, including **tetrazoles**, such as 5-**aminotetrazole**, **tetrazole**, bitetrazole, metal salts of **tetrazoles**; 1,2,4-triazole-5-one, 3-nitro 1,2,4-triazole-5-one and metal salts of triazoles; dicyanamide;. . .

BSUM(11) The . . . serve as an oxidizer, either alone or in combination with other oxidizers such as ammonium, alkali, and alkaline earth metal **nitrates**, chlorates, **peroxides**, and **perchlorates**. Metal

DETD(2) A gas generant formulation of 76.6 wt % CuO, 23.4 wt % 5-**aminotetrazole** (**5AT**) was prepared. Based on the weight of the generant formulation, release agent was added per table 1 below. The

US PAT NO: 5,516,377 L12: 9 of 23

ABSTRACT: A . . . is provided which has at least one salt of 5-nitraminotetrazole and an at least one oxidizer selected from among inorganic **nitrates**, inorganic nitrites, metal oxides, metal **peroxides**, organic **peroxides**, inorganic **perchlorates**, inorganic chlorates, metal hydroxides, and mixtures thereof.

BSUM(18) A . . . inorganic salt of 5-nitraminotetrazole in combination with at least one oxidizer selected from the group consisting of inorganic nitrites, inorganic **nitrates**, metal oxides, metal or organic **peroxides**, inorganic **perchlorates**, inorganic chlorates, metal hydroxides, and mixtures thereof.

BSUM(24) In . . . can produce a lower flame temperature and can provide an improved filterable slag. Oxidizers include metal oxides, metal hydroxides, inorganic **nitrates**, inorganic nitrites, inorganic chlorates, inorganic **perchlorates**, metal or organic **peroxides**, and the like. Exemplary organic **peroxides** include, for instance, di-t-butyl **peroxide**, t-butyl hydroperoxide, benzoyl **peroxide**, or a peracids such as peracetic acid. Exemplary metal **peroxides** include alkaline earth metal **peroxides** (Ca, Ba, Sr, or Mg) and, for instance, transition metal **peroxides**. Persulfates can also be used such as sodium persulfate.

BSUM(25) The . . . combined with a secondary fuel or tertiary fuel. Illustrative of the suitable secondary and tertiary fuels are dicyanamide salts, metal bi-**tetrazole** salts, complexes or salts of **aminotetrazole**, **tetrazoles**, triazoles, ureas, guanidines, nitramine, nitroguanidine and other high nitrogen content

BSUM(31) Illustrative . . . nitrate hexahydrate, and, optionally, a burn rate catalyst such as fumed alumina; zinc nitraminotetrazole, ammonium nitrate, strontium nitrate, and zinc bis-(5-**aminotetrazole**); ammonium nitraminotetrazole, copper(II) oxide; zinc nitraminotetrazole and copper(II) oxide, and a secondary fuel . . .

DETD(26) In a manner analogous to Example 6, pellets were made from a composition of zinc bis-(**aminotetrazole**) (21.16 grams), ammonium nitrate (58.04 grams), zinc nitraminotetrazole dihydrate (20.78 grams). The pellets were combusted, and the burn rate was. . .

CLMS(1) What . . . 5-nitraminotetrazole and an oxidizing effective amount of at least one oxidizer selected from the group consisting of metal oxides, metal **peroxides**, metal hydroxides, and mixture thereof, wherein

CLMS(15) 15. . . . (Merck Index (9th Edition 1976)); an oxidizing effective amount of an oxidizer selected from the group consisting of metal oxides, metal **peroxides**, metal hydroxides, and mixtures thereof; a secondary oxidizer; a secondary fuel selected from the group consisting of a metal dicyanamide, a metal bitetrazole, **tetrazoles**, **aminotetrazoles**. . .

US PAT NO: 5,501,823 L12: 10 of 23

ABSTRACT: A solid composition for generating nitrogen containing gas is provided. The composition includes an oxidizer and a **non**-**azide** fuel selected from anhydrous **tetrazoles**, derivatives, salts, complexes, and mixtures thereof. Preferred **tetrazoles** include 5-**aminotetrazole** and . . .

BSUM(2) The . . . compositions for inflating automobile air bags and similar devices. More particularly, the present invention relates to the use of anhydrous **tetrazole** compounds as a primary fuel

BSUM(15) One group of chemicals that has received attention as a possible replacement for sodium azide includes **tetrazoles** and triazoles. These materials are generally coupled with conventional oxidizers such as KNO.sub.3 and Sr(NO.sub.3).sub.2. Some of the **tetrazoles** and triazoles that have been specifically mentioned include 5-**aminotetrazole**, 3-amino-1,2,4-triazole, 1,2,4-triazole,. . .

BSUM(20) The novel solid compositions of the present invention include a **non**-**azide** fuel and an appropriate oxidizer. Specifically, the present invention is based upon the discovery that improved gas generant compositions are obtained using anhydrous **tetrazoles**, such as 5-**aminotetrazole** and

BSUM(28) Importantly, . . . particularly when exposed to a humid environment. Following pressing of the pellets, the gas generating material is dried until the **tetrazole** is substantially anhydrous. Generally, the **tetrazole** containing composition loses about 3% to 5% of its weight during the drying process. This is

DETD(2) The present invention relates to the use of an anhydrous **tetrazole**, or a salt or a complex thereof, as the primary fuel in a novel gas generating composition.

DETD(4) Other **tetrazoles** within the scope of the present invention include **tetrazole**, 5-**aminotetrazole** (hereinafter sometimes referred to as "**5AT**"), bitetrazole, the n-substituted derivatives of **aminotetrazole** such as

DETD(6) In . . . flame temperature and an improved filterable slag. Such oxidizers include metal oxides and metal hydroxides. Other oxidizers include a metal **nitrate**, a metal nitrite, a metal chlorate, a metal **perchlorate**, a metal **peroxide**, ammonium **nitrate**, ammonium **perchlorate** and the like. The use of metal oxides or hydroxides as oxidizers is particularly useful and such materials include for. . .

DETD(10) **Tetrazoles** within the scope of the present invention are commercially available or can be readily synthesized. With regard to synthesis of. . .

DETD(76) In this example compositions within the scope of the invention were prepared. The compositions comprised 76.6% CuO and 23.4% 5-**aminotetrazole**. In one set of compositions, the 5-**aminotetrazole**

US PAT NO: 5,500,059 L12: 11 of 23

ABSTRACT: A solid composition for generating a nitrogen containing gas is provided. The composition includes an oxidizer and a 5-**aminotetrazole** fuel selected from anhydrous 5-**aminotetrazole** derivatives, salts, complexes, and mixtures thereof. . . .

BSUM(15) One group of chemicals that has received attention as a possible replacement for sodium azide includes **tetrazoles** and triazoles. These materials are generally coupled with conventional oxidizers such as KNO.sub.3 and Sr(NO.sub.3).sub.2. Some of the **tetrazoles** and triazoles that have been specifically mentioned include 5-**aminotetrazole**, 3-amino-1,2,4-triazole, 1,2,4-triazole, 1H-**tetrazole**,

BSUM(20) The novel solid compositions of the present invention include a **non**-**azide** fuel and an appropriate oxidizer. Specifically, the present invention is based upon the discovery that improved gas generant compositions are obtained using substantially anhydrous 5-**aminotetrazole**, or a salt or a complex thereof,

BSUM(23) (a) obtaining a desired quantity of gas generating material, said gas generating material comprising an oxidizer and hydrated 5-**aminotetrazole**;

BSUM(29) Following pressing of the pellets, the gas generating material is dried until the **tetrazole** is substantially anhydrous. Generally, the hydrated 5-**aminotetrazole** composition loses about 3% to 5% of its weight during the drying process. The 5-**aminotetrazole** itself loses about 17% of its weight (theoretical weight loss is 17.5%). This is found to occur, for example, after. . . of the invention, but it is presently preferred that the temperature not exceed 150.degree. C. FIG. 1 illustrates a typical 5-**aminotetrazole**

DETD(3) In . . . lower flame temperature and an improved filterable slag. Such oxidizers include metal oxides and metal hydroxides. Other oxidizers include metal **nitrates**, metal nitrites, metal chlorates, metal **perchlorates**, metal **peroxides**, ammonium **nitrate**, ammonium **perchlorate** and . . .

DETD(5) The . . . in a fuel-effective amount, with an appropriate oxidizing agent to obtain a gas generating composition. In a typical formulation, the **tetrazole** fuel comprises from about 10 to about 50 weight percent of the composition and the oxidizer comprises from about 50. . .

DETD(49) A crystalline sample of **aminotetrazole** hydrate (Dynamit Nobel) was dehydrated at 220.degree. F. losing 17.1% of it original weight (17.5% being theoretical weight loss). A portion of this anhydrous **aminotetrazole** was recrystallized from methanol and an additional portion was recrystallized from ethanol. The resulting solids were heated at 220.degree. F. to a constant weight. Each type of **aminotetrazole** was forced through a 60 mesh sieve. Three compositions containing grd pyro cupric oxide (38.30 g, 76.60%) and **aminotetrazole** (11.70 g, 23.40%) were mixed and processed in the solvent from which the . . .

DETD(50) TABLE 2
Cupric Oxide/**Aminotetrazole** Formulations*

DETD(52) A . . . (11.16 g) grd pyro cupric oxide as described in Example 4, 26.25% (5.25 g) of the 5.6 micron, partially dehydrated **aminotetrazole** (AT 0.8H.sub.2 O) described in Example 3, and 17.96% (3.59

DETD(54) Three gas generating compositions were prepared utilizing the anhydrous 5-**aminotetrazole** powder prepared in Example 3 as the fuel (21.24%, 10.62 g), the three different types of cupric oxide described in. . . to those described in Example 4, 5, and 6. The results are summarized in Table 3. As with the cupric oxide/**aminotetrazole** formulations, burn rate values are dependent on the type of cupric oxide and follow the same trend: ungrd hydro< <grd hydro<grd . . .

DETD(55) TABLE 3
Cupric Oxide/Strontium Nitrate/**Aminotetrazole** Formulations*

DETD(57) A gas generating composition was prepared utilizing anhydrous 5-**aminotetrazole** powder (9.86%, 0.54 g, Fairmont), 8.7 micron ungrd hydro cupric oxide (55.30%, 3.04 g, Aldrich) as . . .

DETD(59) A gas generating composition was prepared utilizing anhydrous 5-**aminotetrazole** powder (12.64%, 1.27 g, Fairmont), 8.7 micron ungrd hydro cupric oxide (31.52%, 3.15 g, Aldrich) as . . .

DETD(60) TABLE 4
Cupric Oxide, **Aminotetrazole** Formulations
Effect of Additives on Burn Rate

CLMS(1) 1. A gas generating composition comprising a fuel selected from the group consisting of anhydrous 5-**aminotetrazole**, anhydrous salts thereof, anhydrous complexes thereof, and mixtures thereof, and an oxidizer, said oxidizer being selected from the group consisting of metal oxides, metal hydroxides, metal **nitrates**, metal nitrites, metal chlorates, metal **perchlorates**, metal **peroxides**, ammonium **nitrate**, ammonium **perchlorate**, and mixtures thereof.

CLMS(10) 10. A gas generating composition as defined in claim 1, . . . 5-**aminotetrazole**

US PAT NO: 5,472,647 L12: 12 of 23

ABSTRACT: A . . . charge wherein the gas generating material is an oxidizer and at least one fuel selected from the group consisting of **tetrazoles**. More particularly, a preferred method involves preparing an anhydrous gas generating composition by preparing a slurry of gas generating material. . . wherein the oxidizer is selected from the group consisting of a metal **peroxide**, an inorganic **nitrate**, an inorganic nitrite, a metal oxide, a metal hydroxide, an inorganic chlorate, an inorganic **perchlorate**, or a mixture thereof, and the fuel is selected from the group consisting of **tetrazoles** . . .

BSUM(11) The method according to the present invention overcomes or minimizes processing difficulties encountered in manufacturing charges, such as pellets, from anhydrous **tetrazole**-fueled gas generant

DETD(20) Other **tetrazoles** include **tetrazole**, 5-**aminotetrazole** (hereinafter sometimes referred to as "**5AT**"), bitetrazole, the n-substituted derivatives of **aminotetrazole** such as

DETD(21) Salts or complexes of any of these **tetrazoles** including those of transition metals such as copper, cobalt, iron, titanium, and zinc; alkali metals such as potassium and sodium;. . .

DETD(22) An . . . include metal oxides and metal hydroxides, such as transition metal oxides and transition metal hydroxides. Other oxidizers include a metal **nitrate** such as, for instance, an alkali metal **nitrate** or strontium **nitrate**, a metal nitrite such as, for instance, an alkali metal nitrite or a nitrite of, for instance, strontium, cobalt or chromium, a metal chlorate such as, for instance, KClO.sub.3, a metal **perchlorate** such as, for instance, NaClO.sub.4, KClO.sub.4 and the like, a metal **peroxide** such as, for instance, an alkaline earth **peroxide**, ammonium **nitrate**, ammonium **perchlorate** and the like. . . .

DETD(23) The **tetrazole** fuel is combined, in a fuel-effective amount, with an appropriate oxidizing agent to obtain a gas generating composition. In a typical formulation, the **tetrazole** fuel comprises from about 10 to about 50 weight percent of the composition and the oxidizer comprises from about 50. . .

DETD(24) Additives . . . the oxides of nitrogen from the combustion products of a gas generant composition, including alkali metal salts and complexes of **tetrazoles**, **aminotetrazoles**, triazoles and related nitrogen heterocycles of which potassium **aminotetrazole**. . .

DETD(41) A **non**-**azide** gas generating composition is prepared by blending 274.8 grams of the **non**-**azide** fuel, BTA monohydrate, having a nominal particle size . . .

US PAT NO: 5,472,534 L12: 13 of 23

ABSTRACT: A . . . non-metallic cation; and an oxidizing effective amount of an inorganic oxidizer. The oxidizer can be an inorganic nitrite, an inorganic **nitrate**, a metal **peroxide**, a metal oxide, a metal hydroxide, an inorganic **perchlorate**, or an inorganic chlorate. A binder can be added, if desired. r. . .

BSUM(10) A **non**-**azide** gas generating composition according to the present invention comprises a fuel effective amount of at least one non-metallic salt of 5-nitrobarbituric acid and an oxidizing effective amount of an inorganic oxidizer selected from among **nitrate**, nitrite, **peroxide**, metal oxide, metal hydroxide, **perchlorate**, and chlorate oxidizers as well as mixtures thereof.

DETD(2) A **non**-**azide** gas generating composition according to the present invention comprises a fuel effective amount of at least one non-metallic salt of 5-nitrobarbituric acid and an oxidizing effective amount of an oxidizer selected from among inorganic **nitrates**, inorganic nitrites, metal oxides, metal hydroxides, inorganic **perchlorates**, inorganic chlorates, inorganic **peroxides**, and mixtures thereof.

DETD(3) A . . . by weight, preferably about 55% to about 78% by weight, of an inorganic oxidizer selected from among nitrite oxidizers, metal **peroxides**, **nitrate** oxidizers, metal oxides, metal hydroxides, **perchlorate** oxidizers, chlorate oxidizers, and mixtures thereof.

DETD(6) The oxidizer is a metal **peroxide**, an inorganic **nitrate**, an inorganic nitrite, a metal oxide, a metal hydroxide, an inorganic chlorate, an inorganic **perchlorate**, or a mixture thereof. Thus, suitable oxidizers include a metal **nitrate**, a metal nitrite, a metal chlorate, a metal **perchlorate**, a metal **peroxide**, ammonium **nitrate**, ammonium **perchlorate** and the like. Suitable oxidizing compounds can have a cation such as, for instance, ammonium, metal ions of metals from. . . Fe.sub.2 O.sub.3, MnO.sub.2, MoO.sub.3, Bi.sub.2 MoO.sub.6, Bi.sub.2 O.sub.3, and Cu(OH).sub.2, can be used. Other exemplary oxidizers include, among others, metal **peroxides** or **nitrates** . . .

DETD(9) Additives . . . the oxides of nitrogen from the combustion products of a gas generant composition, including alkali metal salts and complexes of **tetrazoles**, **aminotetrazoles**, triazoles and related nitrogen heterocycles of which potassium **aminotetrazole**, sodium carbonate and potassium carbonate are exemplary. The composition can also include materials which facilitate the release of the composition. . .

US PAT NO: 5,460,668 L12: 14 of 23

BSUM(16) In accordance with the present invention, the fuel utilized in the **nonazide** gas generant is preferably selected from compounds that maximize the nitrogen content of the fuel and regulate the carbon and. . . content thereof to moderate values. Such fuels are typically selected from azole compounds or metal salts of azole compounds, particularly **tetrazole** compounds such as **aminotetrazole**, **tetrazole**,

BSUM(17) Oxidizers . . . the range of 0.1% to about 5% and preferably from approximately 0.5% to 2%. Typically, oxidizers are chosen from inorganic **nitrates**, nitrites, chlorates or **perchlorates** of alkali metals, alkaline earth metals or ammonium. Strontium and barium **nitrates** are easy to obtain in the anhydrous state and are excellent oxidizers. Strontium **nitrate** and barium **nitrate** are most preferred

BSUM(18) A . . . serves in a dual capacity as a slag former. The most preferred oxidizer which also enhances slag formation is strontium **nitrate**, but barium **nitrate** is also effective. Generally, slag formers may be selected from numerous compounds, such as alkaline earth metal or transition metal oxides, hydroxides, carbonates, oxalates, **peroxides**, **nitrates**, chlorates, and **perchlorates**, or alkaline earth metal salts of **tetrazoles**, bitetrazoles and triazoles, as well as other compounds.

BSUM(19) Another . . . air bags, it is advantageous to use compounds which have a high nitrogen content, such as alkali metal salts of **tetrazoles** or triazoles. These materials serve multiple functions when incorporated into the gas generant because they function as fuels which produce. . .

BSUM(20) The . . . effectively used in a gas generant is quite broad. For example, as little as 2% of the potassium salt of 5-**aminotetrazole** (K5-AT) is effective as an additive, and in cases where the K5-AT also serves as the primary fuel and gas. . . about 2 to about 12% by weight. The alkali metal salts of 5-**aminotetrazole**, **tetrazole**, bitetrazole and 3-nitro-1,2,4- triazole-5-one (NTO) are usable because of their high nitrogen content. Lithium, sodium and potassium are preferred alkali metals, but rubidium and cesium may also be utilized. The most preferred alkali metal salt is the potassium salt of 5-**aminotetrazole**.

BSUM(22) The types of glass that are effective vary depending upon the combustion temperature of a particular **nonazide** fuel and oxidizer. The glass compound utilized is preferably a high-temperature softening glass, because of the aforesaid high temperatures typically exhibited by **nonazide** gas generants.

DETD(3) A mixture of 5-**aminotetrazole** (5-AT), strontium nitrate [Sr(NO.sub.3).sub.2], K5-AT, and powdered PYREX glass brand No. 7740 is prepared having the following composition in. . .

US PAT NO: 5,439,537 L12: 15 of 23

BSUM(15) One group of chemicals that has received attention as a possible replacement for sodium azide includes **tetrazoles** and triazoles. These materials are generally coupled with conventional oxidizers such as KNO.sub.3 and Sr(NO.sub.3).sub.2. Some of the **tetrazoles** and triazoles that have been specifically mentioned include 5-**aminotetrazole**, 3-amino-1,2,4-triazole, 1,2,4-triazole, 1H-**tetrazole** . . .

DETD(12) In addition, small amounts, such as up to about 10 wt. %, of supplemental oxidizing agents, such as metal oxides, **peroxides**, **nitrates**, nitrites, chlorates and **perchlorates**, can, if desired, be combined with a metal hydroxide-containing oxidizer. With the use of **nitrates**, and nitrites as supplemental oxidizing agents, small amounts of nitrogen will be produced in addition to water vapor.

US PAT NO: 5,429,691 L12: 16 of 23

BSUM(15) One group of chemicals that has received attention as a possible replacement for sodium azide includes **tetrazoles** and triazoles. These materials are generally coupled with conventional oxidizers such as KNO.sub.3 and Sr(NO.sub.3).sub.2. Some of the **tetrazoles** and triazoles that have been specifically mentioned include 5-**aminotetrazole**, 3-amino-1,2,4-triazole, 1,2,4-triazole, 1H-**tetrazole** . . .

DETD(20) In addition, small amounts, such as up to about 10 wt. %, of supplemental oxidizing agents, such as metal oxides, **peroxides**, **nitrates**, nitrites, chlorates and **perchlorates**, can, if desired, be combined with the inorganic oxidizer.

US PAT NO: 5,197,758 L12: 17 of 23

ABSTRACT: Gas generating compositions or propellants are provided which comprise a **non**-**azide** fuel which is a transition metal complex of an aminoarazole. Preferred transition metal complexes are zinc and copper complexes of 5-**aminotetrazole** and 3-amino-1,2,4-triazole, with the zinc complexes most preferred. The propellant compositions also include a conventional oxidizer, such as potassium nitrate. . .

DETD(2) The . . . of this invention relates to gas generant or propellant compositions based on transition metal complexes of an aminoarazole as the **non**-**azide** gas producing fuel material. As used herein, the term "aminoarazole" refers to compounds which contain either a **tetrazole** or triazole ring with at least one amino group bonded directly to at least one of the carbon atoms of the **tetrazole** or triazole ring.

DETD(3) The . . . generants over previously employed nitrogen producing materials. First, they avoid the aforementioned disadvantages of the azide compounds. Second, while various **aminotetrazoles** per se are known to be adequate generators of nitrogen gas (see several of the U.S. patents aforementioned),

DETD(6) The . . . an oxidizer for the aminoarazole nitrogen-producing fuel is normally used, which is preferably anhydrous. Such oxidizers include metallic nitrites and **nitrates**, such as KNO.sub.3 and Sr(NO.sub.3).sub.2, and various oxides sulfides, iodides, **perchlorates**, chromates, **peroxides**

DETD(22) This example illustrates the preparation of a transition metal of an aminoarazole, i.e., a zinc complex of 5-**aminotetrazole**, Zn(AT).sub.2.

DETD(23) 17.0 g of 5-**aminotetrazole** (AT) in hot water was added to 200-300 ml of water. . . .

DETD(26) This example illustrates the preparation of a transition metal complex of an aminoarazole, i.e., a copper (II) complex of 5-**aminotetrazole**, Cu(AT).sub.2.

DETD(50) These data indicate similar flame temperatures and burning rates are obtained with aminotriazole complexes relative to those prepared with **aminotetrazole** as described in Example 5. Furthermore, burning rate is increased by the use of potassium nitrate rather than strontium nitrate. . .

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BSUM(3) This . . . More particularly, this invention relates to improved gas generant compositions including a fuel for producing the nitrogen rich gas, especially **non**-**azides**, and a novel oxidizer therefor comprising an inorganic compound having a **poly(nitrito) transition metal complex anion.**

BSUM(15) A number of approaches to a **non**-**azide** nitrogen gas generant have been investigated in the prior art as disclosed, for example, in U.S. Pat. Nos. 3,055,911; 3,348,985;. . . which are oxidized to produce non-corrosive and, often termed, "non-toxic" gases. However, there some disadvantages to the use of such **non**-**azide** fuel materials. For example, the gas products produced from burning these type fuels sometimes produce unacceptably high levels of carbon. . .

BSUM(16) Typical oxidizers conventionally used in prior art gas generant compositions are: (1) ammonium **nitrate**, and alkali and alkaline earth metal nitrite and **nitrates**, such as KNO.sub.3 and Sr(NO.sub.3).sub.2, (2) ammonium oxalate and various metallic oxides, mixed oxides and bi-metallic complex oxides, based on. . . including fluorides, chlorides and iodides, such as FeF.sub.3 and CrCl.sub.3, as well as

various organic chlorides and iodides, (4) ammonium **perchlorate**, and alkali metal chlorates and **perchlorates**, such as KClO.sub.4, (5) various inorganic sulfides, such as MoS2, and sulfur and (6) various other inorganic **peroxides**, permanganates, chromates and dichromates; as exemplified in U.S. Pat. Nos.

DETD(5) A . . . which generants nitrogen-containing gas, which may be either an azide, for example NaN.sub.3, as is well known; or preferably a **non**-**azide**, the most preferred of which is potassium diliturate (KDL), C.sub.4 H.sub.2 N.sub.3 OK. Examples of other **non**-**azide** fuels are bis(5-**aminotetrazole**)Zn (ZnAT), C.sub.2 H.sub.4 N.sub.10 Zn, and potassium nitroorotate,

DETD(11) The . . . the natural complex as a precipitate. See Example 4. Other alkali and alkaline earth metal salts, such as the acetates, **perchlorates**, **nitrates** or tetrafluoroborates, may also be used so long as the anion does not displace the nitrito moieties of the transition. . .

DETD(24) B. In the following example, the bis (5-**aminotetrazole**) complex of zinc (ZnAT), ZnC.sub.2 H.sub.4 N.sub.10, was employed as the **non**-**azide** gas generating fuel with the novel oxidizer. Burning rates are reported at 1000 psi in inches per second.

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BSUM(16) An . . . and Inflation of Gas Bags Therewith". This patent describes use of alkali or alkaline earth metal salts of a hydrogen-free **tetrazole** compound and oxidizers of sodium nitrate, sodium nitrite and potassium nitrate or alkaline earth nitrates. A filter design is disclosed. . .

BSUM(32) U.S. . . . Crash Bag" discloses the use of alkali metal salts, alkaline earth metal salts or ammonium salt of a hydrogen containing **tetrazole** in the range of about 20 to about 65 wt. %. . . .

BSUM(42) **Tetrazole** compounds such as **aminotetrazole**, **tetrazole**, bitetrazole and metal salts of these compounds as well as triazole compounds such as 1,2,4-triazole-5-one or . . .

BSUM(44) Oxidizers . . . they are the preferred method of including a high-temperature slag former into the reaction system. The alkaline earth and cerium **nitrates** are all oxidizers with high-temperature slag forming potential, although most of these salts are hygroscopic and are difficult to use effectively. Strontium and barium **nitrates** are easy to obtain in the anhydrous state and are excellent oxidizers. Alkali metal **nitrates**, chlorates and **perchlorates** are other useful oxidizers when combined with a high-temperature slag former.

BSUM(46) Metal salts as fuels, such as the calcium or strontium salt of 5-**aminotetrazole**, **tetrazole**, or ditetrazole are also useful high-temperature slag formers, although not as efficient as the oxidizers.

BSUM(57) a. A fuel selected from the group of **tetrazole** compounds consisting of **aminotetrazole**, **tetrazole**, bitetrazole and metal salts of these compounds as well as triazole compounds

BSUM(58) b. An oxygen containing oxidizer compound selected from the group consisting of alkali metal, alkaline earth metal, lanthanide and ammonium **nitrates** and **perchlorates** or from the group consisting of alkali metal or alkaline earth metal chlorates or **peroxides**.

BSUM(59) c. . . . temperature slag forming material selected from the group consisting of alkaline earth metal or transition metal oxides, hydroxides, carbonates, oxalates, **peroxides**, **nitrates**, chlorates and **perchlorates** or from the group consisting of alkaline earth metal salts of **tetrazoles**, bitetrazoles

BSUM(60) d. . . . consisting of silicon dioxide, boric oxide and vanadium pentoxide or from the group consisting of alkali metal silicates, borates, carbonates, **nitrates**, **perchlorates** or chlorates or from the group consisting of alkali metal salts of **tetrazoles**, bitetrazoles and triazoles or

BSUM(61) In . . . fuel and the high-temperature slag forming material may be selected from the group

consisting of alkaline earth metal salts of **tetrazoles**, bitetrazoles and triazoles. Both the oxygen containing oxidizer compound and high-temperature slag forming material may be comprised of one or more of the group consisting of alkaline earth metal and lanthanide **nitrates**, **perchlorates**, chlorates and **peroxides**. Both the fuel and the low-temperature slag forming material may comprise one or more of the group consisting of alkali metal salts of **tetrazoles**, bitetrazoles and triazoles. Both the oxygen containing oxidizer compound and the low-temperature slag forming material may comprise one or more of the group consisting of alkali metal **nitrates**, **perchlorates**, chlorates and **peroxides**.

BSUM(62) The fuel may comprise 5-**aminotetrazole** which is present in a concentration of about 22 to about 36% by weight, where the oxygen containing oxidizer compound. . .

BSUM(63) Alternatively, the fuel and high-temperature slag forming material may comprise the strontium salt of 5-**aminotetrazole** which is present in a concentration of about 30 to about 50% by weight . . .

BSUM(64) Another combination comprises the 5-**aminotetrazole** which is present in a combination of about 22 to about 36% by weight, where the oxygen containing oxidizer compound. . .

BSUM(65) Yet another combination comprises the potassium salt of 5-**aminotetrazole** which is present in a concentration of about 2 to about 30% by weight which serves in part as a fuel and in part as a low-temperature slag former and wherein 5-**aminotetrazole** in a concentration of about 8 to about 40%

BSUM(70) Another preferred composition is one wherein the fuel comprises 5-**aminotetrazole** in a concentration of about 26 to about 32% by weight, the oxygen containing oxidizer compound comprises strontium nitrate in a concentration of about 52 to about 58% by weight,

BSUM(71) Still another preferred composition is one wherein the fuel comprises 5-**aminotetrazole** in a concentration of about 26 to about 32% by weight, the oxygen containing oxidizer compound comprises strontium nitrate in a concentration of about 52 to about 58% by weight, the chemical additive comprises the potassium salt of 5-**aminotetrazole** in a concentration of about 2 to about 12% by weight . . .

BSUM(74) The . . . automobile airbags it is advantageous to use compounds which have a high nitrogen content such as alkali metal salts of **tetrazoles** or triazoles. These materials serve multiple functions when incorporated into a gas generant. In addition to reducing the amount of. . .

DETD(19) . . .
1. **5AT** (22-36)
Sr(NO.sub.3).sub.2
SiO.sub.2
SrO
Sr(NO.sub.3).sub.2
(38-62) (2-18) SrCO.sub.3
SiO.sub.2 SrSiO.sub.3
2. **5AT** (22-36)
Sr(NO.sub.3).sub.2
Clay SrO
Sr(NO.sub.3).sub.2
(38-62) (2-18) SrCO.sub.3 . . .

DETD(21) A mixture of 5-**aminotetrazole** (5 AT), strontium nitrate (SrN) and bentonite clay was prepared having the following composition in percent by weight: 33.1% 5. . .

DETD(27) A mixture of 5 AT, SrN, clay and the sodium salt of **tetrazole** (NaT) was prepared having the following composition in percent by weight: 30.4% 5 AT, 54.2% SrN, 7.4% clay and 8.0%. . .

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BSUM(18) Use of triazole and **tetrazole** reactants (U.S. Pat. Nos. 4,948,439 and 4,931,112) and metal nitrides (U.S. Pat. No. 4,865,667) have also been attempted, however, none. . .

BSUM(36) "An", having strong oxidizing properties and comprising one of the group consisting of a **nitrate**, nitrite, **perchlorate**, chlorate, chromate, dichromate, manganate, permanganate and perborate

CLMS(2) 2. . . . propellant of claim 1 wherein the oxidizer salt is at least one member selected from . . . sodium **nitrate**, potassium **nitrate**, sodium **perchlorate**, and potassium **perchlorate**.

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BSUM(26) The primary advantage of a new **nonazide** gas generant composition in accordance with the instant invention is that solid combustion products are easily filtered from the gas produced. The **nonazide** gas generant uses **tetrazoles** or **tetrazole** salts as the fuel . . .

BSUM(33) **Tetrazole** compounds such as **aminotetrazole**, **tetrazole**, bitetrazole and metal salts of these compounds, as well as triazole compounds such as 1,2,4-triazole-5-one or 3-nitro 1,2,4-triazole-5-. . .

BSUM(34) It . . . of these compounds can function, at least in part, as high temperature slag formers. For example, the calcium salt of **tetrazole** or bitetrazole forms, upon combustion, calcium oxide . . .

BSUM(35) Oxidizers . . . they are the preferred method of including a high-temperature slag former into the reaction system. The alkaline earth and cerium **nitrates** are all oxidizers with high-temperature slag forming potential, although most of these salts are hygroscopic and are difficult to use effectively. Strontium and barium **nitrates** are easy to obtain in the anhydrous state and are excellent oxidizers. Alkali metal **nitrates**, chlorates and **perchlorates** are other useful oxidizers when combined with a high-temperature slag former.

BSUM(37) Metal salts as fuels, such as the calcium or strontium salt of 5-**aminotetrazole**, **tetrazole**, or ditetrazole are also useful high-temperature slag formers, although not as efficient as the oxidizers.

BSUM(48) a. A fuel selected from the group of **tetrazole** compounds consisting of **aminotetrazole**, **tetrazole**, bitetrazole and metal salts of

BSUM(49) b. An oxygen containing oxidizer compound selected from the group consisting of alkali metal, alkaline earth metal, lanthanide and ammonium **nitrates** and **perchlorates** or from the group consisting of alkali metal or alkaline earth metal chlorates or **peroxides**.

BSUM(50) c. . . . temperature slag forming material selected from the group consisting of alkaline earth metal or transition metal oxides, hydroxides, carbonates, oxalates, **peroxides**, **nitrates**, chlorates and **perchlorates** or from the group consisting of alkaline earth metal salts of **tetrazoles**,

BSUM(51) d. . . . consisting of silicon dioxide, boric oxide and vanadium pentoxide or from the group consisting of alkali metal silicates, borates, carbonates, **nitrates**, **perchlorates** or chlorates or from the group consisting of alkali metal salts of **tetrazoles**, bitetrazoles and triazoles or from the group consisting of the various naturally occurring clays and talcs.

BSUM(52) In . . . fuel and the high-temperature slag forming material may be selected from the group consisting of alkaline earth metal salts of **tetrazoles**, bitetrazoles and triazoles. Both the oxygen containing oxidizer compound and high-temperature slag forming material may be comprised of one or more of the group consisting of alkaline earth metal and lanthanide **nitrates**, **perchlorates**, chlorates and **peroxides**. Both the fuel and the low-temperature slag forming material may comprise one or more of the group consisting

of alkali metal salts of **tetrazoles**, bitetrazoles and triazoles. **Both the xygen containing oxidizer compound and the low-temperature slag forming material may comprise one or more of the group consisting of alkali metal **nitrates**, **perchlorates**, chlorates and **peroxides**.**

BSUM(55) Another combination comprises the 5-**aminotetrazole** which is present in a combination of about 22 to about 36% by weight, . . .

BSUM(56) Yet another combination comprises the potassium salt of 5-**aminotetrazole** which is present in a concentration of about 2 to about 30% by weight which serves in part as a. . .

DETD(19) . . .
1. **5AT**(22-36)
Sr(NO.sub.3).sub.2
SiO.sub.2
SrO
Sr(NO.sub.3).sub.2
(38-62) (2-18) SrCO.sub.3
SiO.sub.2 SrSiO.sub.3
2. **5AT**(22-36)
Sr(NO.sub.3).sub.2
Clay SrO . . .

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BSUM(8) Several . . . alkali metal azide or alkaline earth azide, an alkali metal oxidant and an nitrogeneous compound such as an amide or **tetrazole**, and silica as an optional additive.

BSUM(12) U.S. . . . to a gas generating composition consisting of an alkali or alkaline earth metal azide, an oxidizing compound such as a **peroxide**, **perchlorate**, or **nitrate**, an oxide such as

L13 2 L11 NOT L12

1. 5,525,170, Jun. 11, 1996, Fumaric acid-based gas generating compositions for airbags; Armin Stark, et al., 149/85, 77, 83

2. 5,401,340, Mar. 28, 1995, Borohydride fuels in gas generant compositions; Dan W. Doll, et al., 149/22, 109.2; 280/728.1

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BSUM(6) In . . . mixtures have been proposed (U.S. Pat. No. 4,948,439) containing as their principal component organic compounds rich in nitrogen such as **tetrazoles** or **tetrazole** derivates or tetrazolates.
DETD(5) Finally, . . . (substance mixture 2) was compared to a further known propellant mixture (substance mixture 1) consisting of 30.8% (by weight) 5-amino-**tetrazole**, 36.1% (by weight) sodium nitrate . . .

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BSUM(15) One group of chemicals that has received attention ... includes **tetrazoles** and triazoles. . . .
DETD(10) In some cases, supplemental oxidizers such as alkali, alkaline earth, or ammonium perchlorates, chlorates, and **peroxides** in amounts up to about 40%. . .

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